

Argonne National Laboratory

EXAMINATION OF IRRADIATED Ag-In-Cd ALLOYS

by

C. F. Reinke

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C. F. Reinke

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TABLE OF CONTENTS

	<u>Page</u>
ABSTRACT	4
INTRODUCTION	4
FABRICATION	5
Extruded Cast Specimens	6
Extruded Powder Specimens	6
IRRADIATION	6
RESULTS AND DISCUSSION	8
Capsule Disassembly and Visual Examination	8
Dimensional Measurements	9
Tensile Tests	10
Grain Size and Hardness Measurements	12
Metallographic Examination	14
Burnup Analysis	14
CONCLUSIONS	16
ACKNOWLEDGMENTS	16
REFERENCES	17
Irradiation Experiments on Metallurgy	18
Room-Temperature Tensile Properties of Irradiated Ag-15w/25w-3w/0Cd Alloy Specimens	18
VLE DPH Hardness and Grain Size of Ag-15w/25w-3w/0Cd Alloy Specimens	18
VLE Room-Temperature Tensile Properties of Irradiated Ag-15w/25w-3w/0Cd Alloys	18

LIST OF FIGURES

<u>No.</u>	<u>Title</u>	<u>Page</u>
1.	Pictorial Drawing of Specimens and Capsule Components	7
2.	Carbon-Steel Spacer Showing Outline of Ag-In-Cd Specimens . .	7
3.	Ag-In-Cd Specimen after Irradiation	9
4.	Dimensional Code Used for Reporting Specimen Measurements	10
5.	Separation of Nickel Plating from Base Alloy on Extruded Cast Tensile Specimen	12
6.	Cracks in Nickel Plating on Extruded Powder Tensile Specimen.	12
7.	Comparison of Pre- and Postirradiation Microstructures on Ag-15w/oIn-5w/oCd Alloys	15

LIST OF TABLES

<u>No.</u>	<u>Title</u>	<u>Page</u>
I.	Specimen Data	5
II.	Specimen Irradiation History	8
III.	Pre- and Postirradiation Dimensions on Metallographic Specimens	9
IV.	Pre- and Postirradiation Dimensions on Tensile Specimens. . .	10
V.	Room-temperature Tensile Properties of Irradiated Ag-15w/oIn-5w/oCd Alloy Specimens	11
VI.	DPH Hardness and Grain Size of Ag-15w/oIn-5w/oCd Alloy Specimens	12
VII.	Knoop Hardness Survey of Irradiated Ag-15w/oIn-5w/oCd Alloys	13

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ABSTRACT

Extruded cast and extruded powder specimens of Ag-15w/oIn-5w/oCd alloy, plated with 25 μ of nickel or 25 μ each of copper plus nickel, were irradiated for 47 effective full-power days at approximately 300°C in a lattice position of the Engineering Test Reactor. The extruded cast alloys exhibited 0.2% yield strengths, ranging from 114 to 140 kg/cm² in the unirradiated condition, and 96 to 150 kg/cm² after irradiation. The percent elongation and reduction in area in both cases normally ranged from 50 to 60%. The 0.2% yield strength for the extruded powder alloys ranged from 190 to 228 kg/cm² in the unirradiated condition, and 214 to 242 kg/cm² after irradiation. There was a noticeable improvement in both the percent elongation and reduction in area of the extruded powder specimens, the postirradiation values being approximately twice the unirradiated values of 10%.

Recrystallization and grain growth occurred in all specimens during irradiation. The ASTM grain size on the extruded cast specimens changed from 7 to 4-5.5. Hardness measurements of the extruded cast specimens indicated that softening had occurred. Unirradiated material had DPH values ranging from 55 to 76, with corresponding values after irradiation of 36 to 52.

Metallographic examinations indicated that the microstructures had remained a single phase during irradiation.

INTRODUCTION

At present, the only elements that have been used extensively as the absorber material in power-reactor control rods are boron and hafnium. Neither material is ideal, but for different reasons. The main disadvantage of boron is the $B^{10}(n, \alpha)Li^7$ reaction, which introduces two new atoms into the

lattice. Each atom is larger, and its properties differ from those of the parent boron atom and the matrix in which the boron is usually incorporated. For high-flux, long-life cores, radiation damage can be expected to be significant, and thus the suitability of boron and boron-containing materials is questionable. The use of hafnium produces a rod possessing desirable properties and long life. However, the drawback to the use of hafnium is that it is not readily available and is expensive. Considerable time and effort have been devoted to the development of low-cost, readily-available materials that would combine long-time reactivity worth, good mechanical strength and ductility, irradiation stability, and corrosion resistance. One such potential material which has received attention is the ternary alloy of Ag-15w/oIn-5w/oCd.^(1,2) This report presents information on the stability and change in mechanical and physical properties of nickel and copper-plus-nickel plated specimens of the alloy after an irradiation of 47 effective full-power days at approximately 300°C in a lattice position of the Engineering Test Reactor. Specimen fabrication and irradiation were conducted by the WAPD Division of Westinghouse Electric Corporation. The postirradiation examination and evaluation were conducted at the Lemont site of Argonne National Laboratory.

FABRICATION

The fabrication procedures discussed in some detail in reference 1 are believed to be representative of those used to produce the test specimens. The basic steps, obtained from the reference, are outlined below for both the extruded cast and extruded powder alloys used in the irradiation studies. The data are tabulated in Table I.

Table I
SPECIMEN DATA

Specimen Series	Fabrication History	Preparation of Surface for Plating	Type of Plating ^(a)	Vacuum Anneal after Plating
CR-6-A	Induction-melted, cast, and extruded at 600°C.	Cleaned	Ni	1 hr at 550°C
CR-6-B	Induction-melted, cast, and extruded at 600°C.	Etched	Ni	1 hr at 550°C
CR-6-C&D	Induction-melted, cast, and extruded at 600°C.	Etched	Cu and Ni	1 hr at 550°C
PCR-4-E	Induction-melted, atomized, hot-compacted, and extruded at 700°C.	Etched	Ni	2 hr at 600°C
PCR-4-H	Induction-melted, atomized, hot-compacted, and extruded at 700°C.	Etched	Cu and Ni	2 hr at 600°C

^(a)Ni plate was 25 μ thick. Copper plus nickel plate were each 25 μ thick.

Extruded Cast Specimens

The alloys were prepared by induction melting in graphite crucibles under a protective covering of calcined coke. The ingots were bottom-poured directly from the melting crucibles into graphite molds. Just before extrusion, the ingots were homogenized for 4 hr at 600°C. The test specimens were cut from full-size, extruded, control rods and machined to final dimensions. The machined specimens were then chemically etched and plated with either 25 μ of nickel, or 25 μ each of copper plus nickel. After plating, the specimens were given a 1-hr vacuum anneal at 550°C. The purpose of the anneal was to recrystallize the nickel deposit, provide metallurgical bonding between the plate and the base alloy, and enhance the ductility of the nickel plate.

Extruded Powder Specimens

Material of the desired chemical composition was prepared by melting in a graphite crucible under a protective covering of calcined coke. The melt was converted to powder by atomizing the molten alloy in a high-velocity water spray. Each powder particle thus produced had a very thin and continuous oxide film on its surface. The powder was hot-compacted in graphite molds at 600°C and extruded at 700°C to nearly 100% of theoretical density. The test specimens were cut from full-size control rods and machined to final dimensions. The machined specimens were then chemically etched and plated with either 25 μ of nickel, or 25 μ each of copper plus nickel. After plating, the specimens were given a 2-hr anneal at 600°C. The purpose of the anneal was the same as in the case of the extruded cast specimens. The very fine and uniformly-distributed dispersion of the oxide particles improved the mechanical strength of the alloy.

IRRADIATION

Five capsules were irradiated, each containing two tensile and eight metallographic specimens. The test specimens were placed between two slotted halves of a carbon-steel spacer, which was then fastened at each end with screws. Figure 1 is a pictorial drawing of the specimens and the capsule components. The assembled carbon-steel spacer containing the specimens was forced into an aluminum capsule having an interference fit, after which the capsule was welded shut in air. The soundness of the final closure weld was determined by externally pressurizing each capsule to 17.5 kg/cm² with helium and then immersing it in alcohol to check for leaks. As a final check, the capsules were corrosion-tested for 3 $\frac{1}{2}$ days at 170°C.

The capsules were irradiated in the F-8 lattice position of the Engineering Test Reactor in Idaho from October 5, 1958 to December 29, 1958, for a total of 47 effective full-power days. (The Engineering Test Reactor operates at a power level of 175 MW.) Since the irradiation capsules were

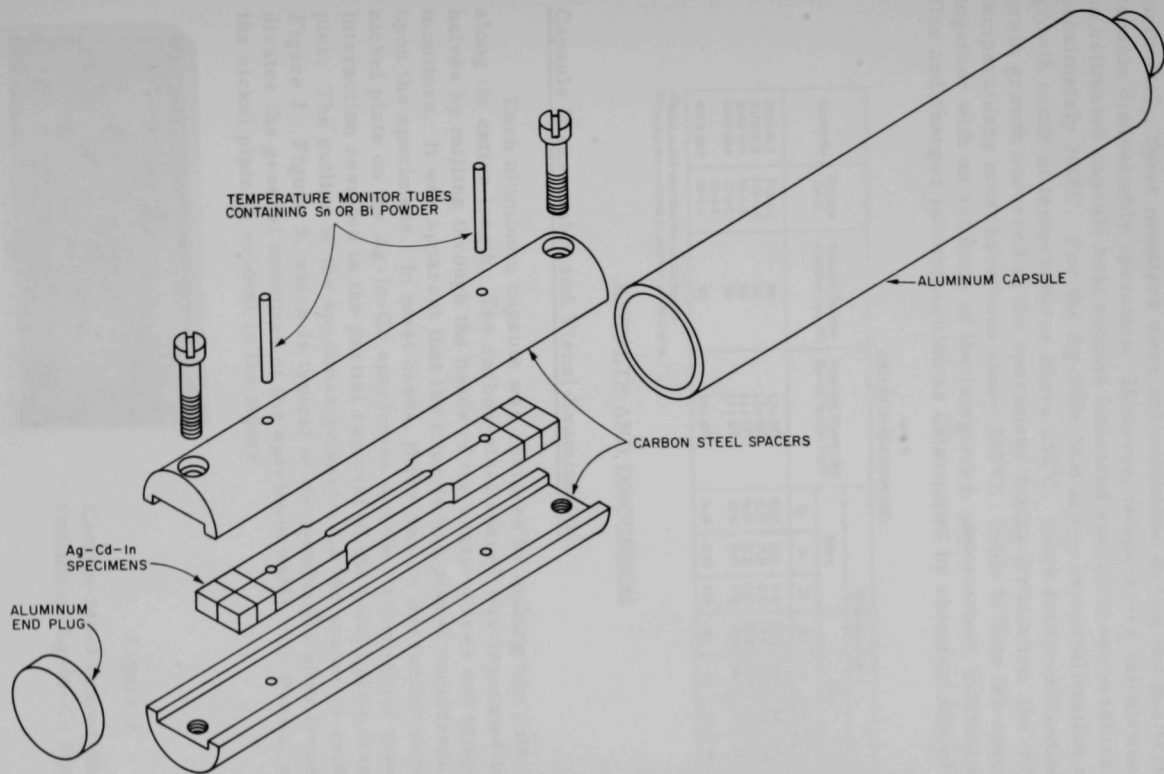


Figure 1. Pictorial Drawing of Specimens and Capsule Components

not instrumented with thermocouples, temperature monitors were included. The monitors consisted of metal powders with known melting points. Unfortunately, these monitors were not recovered during the postirradiation capsule disassembly operation. However, temperature calculations based on estimated capsule heat outputs indicated specimen temperatures of approximately 300°C. For the Ag-15In-5Cd alloy, recrystallization and grain growth occur at temperatures above 200°C. Since recrystallization and grain growth occurred in the specimens during irradiation, the irradiation temperatures must have been above 200°C. Table II lists the specimens, together with an estimate of the integrated, unperturbed, thermal neutron flux and changes in composition as determined by chemical analysis.

Table II
SPECIMEN IRRADIATION HISTORY

Capsule No.	Specimen Series	Estimated Specimen Irradiation Temp, °C	Integrated, Unperturbed, Thermal Neutron Flux ^(a)	Composition, w/o							
				Before				After			
				Ag	In	Cd		Ag	In	Cd	Sn
WAPD 44-1	PCR-4-E	300	1.2×10^{21}	80.23	15.11	4.66		79.23	14.56	5.66	0.55
WAPD 44-2	PCR-4-H	300	1.5×10^{21}	80.75	15.16	4.09		78.36	14.72	6.48	0.44
WAPD 44-3	CR-6-B	300	1.7×10^{21}	80.47	14.86	4.67		78.36	13.36	6.79	1.49
WAPD 44-4	CR-6-C	300	1.6×10^{21}	80.08	14.82	5.10		78.65	14.25	6.53	0.57
WAPD-44-5	CR-6-D CR-6-A	300	1.0×10^{21}	80.65	14.72	4.63		(b)	(b)	(b)	(b)

^(a)Based on ETR-advised flux for the position.

^(b)The analyses contained an unknown source of error.

RESULTS AND DISCUSSION

Capsule Disassembly and Visual Examination

Each aluminum capsule was removed by making two cuts, 180° apart, along its entire length. The carbon-steel spacer was separated into its two halves by milling through the heads of the brass screws and temperature monitors. It was apparent that the capsule had placed considerable restraint upon the specimens. In most cases, there was an interaction between the nickel plate on the Ag-In-Cd specimens and the carbon-steel spacer. This interaction resulted in the partial removal of the nickel plate from the samples. The outline of the specimen geometry on the spacer is evident in Figure 2. Figure 3, which is typical of the specimens after irradiation, indicates the general condition of the specimen surfaces. Partial removal of the nickel plate is evident in the figure.



Figure 2

Carbon-Steel Spacer Showing
Outline of Ag-In-Cd Specimens



Figure 3

Ag-In-Cd Specimen after Irradiation.
Note partial removal of nickel plating.

26778

10X

Dimensional Measurements

Tables III and IV compare the pre- and postirradiation dimensions of the specimens from capsules WAPD-44-3 and WAPD-44-5. Figure 4 shows the locations at which the measurements were made. Since the precision of the measurements was 25μ , the data indicate that no measurable change had occurred in the samples. This was not surprising, as the carbon-steel spacer and aluminum capsule placed considerable restraint upon the specimens. Because of this restraint, the significance of the dimensional data was questionable. Since this would also be true of the dimensional data from the other capsules, no further dimensional measurements were made.

Table III
PRE- AND POSTIRRADIATION DIMENSIONS ON METALLOGRAPHIC SPECIMENS

Capsule	Series	Sample No.	Dimensions, cm ^(a)								
			A			B			C		
			Pre	Post	Δ	Pre	Post	Δ	Pre	Post	Δ
WAPD 44-3	CR-6-B	B-1	0.775	0.777	-	0.576	0.574	-	0.653	0.655	-
		B-2	0.775	0.775	-	0.574	0.576	-	0.650	0.653	-
		B-3	0.775	0.775	-	0.579	0.576	-	0.653	0.653	-
		B-4	0.772	0.772	-	0.576	0.574	-	0.653	0.650	-
		B-7	0.772	0.772	-	0.576	0.574	-	0.653	0.653	-
		B-8	0.772	0.770	-	0.574	0.572	-	0.650	0.648	-
		B-9	0.775	0.775	-	0.576	0.574	-	0.653	0.650	-
		B-10	0.772	0.772	-	0.576	0.576	-	0.650	0.650	-
	CR-6-A	A-1	0.762	0.764	-	0.576	0.576	-	0.645	0.645	-
		A-2	0.762	0.764	-	0.579	0.576	-	0.650	0.648	-
		A-3	0.759	0.759	-	0.572	0.572	-	0.640	0.638	-
		A-4	0.762	0.762	-	0.582	0.576	-0.006	0.645	0.645	-
		A-7	0.757	0.759	-	0.576	0.576	-	0.645	0.645	-
		A-8	0.754	0.752	-	0.566	0.566	-	0.638	0.635	-
		A-9	0.757	0.759	-	0.579	0.579	-	0.648	0.648	-
		A-10	0.757	0.757	-	0.569	0.572	-	0.638	0.638	-

(a) See Figure 4 for dimensional code used for reporting sample measurements. The preirradiation measurements were taken with a ball micrometer and are accurate to ± 0.0013 cm.

The postirradiation measurements were taken with a ball micrometer and are accurate to ± 0.003 cm.

Table IV
PRE- AND POSTIRRADIATION DIMENSIONS ON TENSILE SPECIMENS

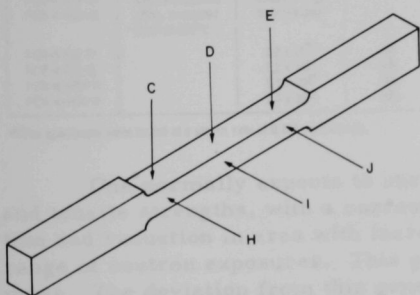
Capsule No.	Series	Sample No.	Dimensions, cm ^(a)						
			Location						
				C	D	E	H	I	J
WAPD 44-3	CR-6-B	B-5	Pre	0.478	0.478	0.478	0.569	0.569	0.566
			Post	0.478	0.475	0.475	0.569	0.569	0.569
			Δ	-	-	-	-	-	-
		B-6	Pre	0.480	0.478	0.478	0.569	0.569	0.569
			Post	0.478	0.475	0.475	0.566	0.566	0.566
			Δ	-	-	-	-	-	-
WAPD 44-5	CR-6-A	A-5	Pre	0.467	0.462	0.465	0.561	0.561	0.561
			Post	0.465	0.460	0.465	0.559	0.559	0.559
			Δ	-	-	-	-	-	-
		A-6	Pre	0.460	0.460	0.462	0.556	0.556	0.554
			Post	0.457	0.460	0.462	0.554	0.554	0.554
			Δ	-	-	-	-	-	-

(a) See Figure 4 for dimensional code used for reporting sample measurements. The preirradiation measurements were taken with a ball micrometer and are accurate to ± 0.0013 cm.

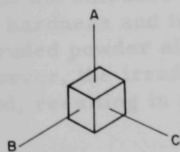
The postirradiation measurements were taken with a ball micrometer and are accurate to ± 0.003 cm.

Tensile Tests

The results of the pre- and postirradiation tensile tests are tabulated in Table V. Since it was known that the mechanical properties of the extruded, coarse-grained specimens varied with the strain rate, all tensile tests were made at a constant strain rate of 0.127 cm/min. The 0.2% yield and ultimate tensile strength of both the extruded cast and extruded powder alloys did not change appreciably under the conditions of the irradiation. The extruded cast specimens exhibited 0.2% yield strengths and ultimate tensile strengths of 114 to 140 kg/cm² and 271 to 324 kg/cm², respectively, in the unirradiated condition with corresponding values after irradiation of 96 to 150 kg/cm² and 283 to 302 kg/cm². The percent elongation and reduction in area in both cases normally ranged from about 50 to 60%. The 0.2% yield strength of the extruded powder specimens was higher than the extruded cast alloys and ranged from 190 to 228 kg/cm² in the unirradiated



TENSILE SPECIMEN



METALLOGRAPHIC SPECIMENS

106-7799

Figure 4. Dimensional Code Used for Reporting Specimen Measurements

condition, and from 214 to 242 kg/cm² after irradiation. The ultimate tensile strengths of the extruded cast and extruded powder specimens were comparable in both the unirradiated and irradiated condition. Ultimate tensile strengths of the extruded powder specimens ranged from 273 to 337 kg/cm²

before irradiation, and from 292 to 325 kg/cm² after irradiation. There was a noticeable improvement in the percent elongation and reduction in area of the extruded powder specimens, the postirradiation values being approximately twice the unirradiated values of 10%.

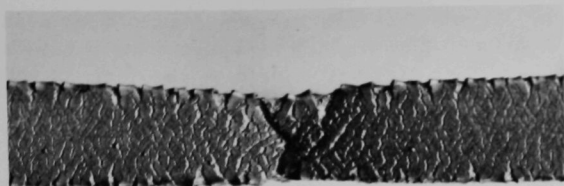
Table V
ROOM-TEMPERATURE TENSILE PROPERTIES OF IRRADIATED Ag-15w/0.1n-5w/0Cd ALLOY SPECIMENS

Designation	Fabrication History	Integrated, Unperturbed, Thermal Neutron Flux	Irradiated Temp, °C	0.2% Yield ^(a) Strength, kg/cm ²	Ultimate ^(a) Tensile Strength, kg/cm ²	Elongation, %	Reduction in Area, %
CR-6-A(A-5)	Extruded cast alloy, annealed 1 hr at 550°C	Unirradiated	-	137	292	45	45
CR-6-B(B-5)		Unirradiated	-	120	271	56	51
CR-6-D(D-5)		Unirradiated	-	114	300	55	56
CR-6-D(D-6)		Unirradiated	-	140	324	56	50
CR-6-A(A-5)		1.0 x 10 ²¹	300	114	288	62	53
CR-6-A(A-6)		1.0 x 10 ²¹	300	121	283	58	56
CR-6-B(B-5)		1.7 x 10 ²¹	300	96	289	66	57
CR-6-B(B-6)		1.7 x 10 ²¹	300	131	301	58	57
CR-6-C(C-5)		1.6 x 10 ²¹	300	150	299	62	54
CR-6-D(D-6)		1.6 x 10 ²¹	300	122	302	59	54
PCR-4-E(A-5)	Extruded powder alloy, annealed 2 hr at 600°C	Unirradiated	-	190	273	11	9
PCR-4-H(D-6)		Unirradiated	-	228	337	13	9
PCR-4-E(E-5)		1.2 x 10 ²¹	300	214	318	17	24
PCR-4-E(E-6)		1.2 x 10 ²¹	300	242	325	19	30
PCR-4-H(H-5)		1.5 x 10 ²¹	300	225	292	23	22
PCR-4-H(H-6)		1.5 x 10 ²¹	300	226	307	20	14

^(a)The specimens were tested at a strain rate of 0.127 cm/cm-min.

One normally expects to observe a marked increase in both yield and tensile strengths, with a corresponding decrease in the percent elongation and reduction in area with increasing neutron exposure over a wide range of neutron exposures. This general trend did not hold for these specimens. The deviation from this general trend was thought to be due to recrystallization during irradiation, thus annealing-out any induced hardening, and in some cases even softening the alloy below the unirradiated condition. In the case of the extruded cast alloys, this was substantiated by the general decrease in hardness and increase in grain size of the irradiated specimens. For the extruded powder alloys, the hardness values did not change appreciably. However, the irradiated microstructures indicated recrystallization had occurred, resulting in at least a partial loss of the oriented as-fabricated structure.

The nickel plating on the tensile specimens cracked during testing. It was most pronounced on the extruded cast specimens. As shown in Figure 5, the plating not only cracked but also separated from the base alloy at the edges of the specimen. Figure 6 is typical of the extruded powder specimens. The nickel plate exhibited considerably less cracking and separation from the base alloy. No difference in behavior was noted between the plating on the irradiated and unirradiated specimens. The difference in behavior between the plating on the extruded cast and extruded powder alloys was believed to be due to the greater percent elongation and reduction in area exhibited by the extruded cast specimens.



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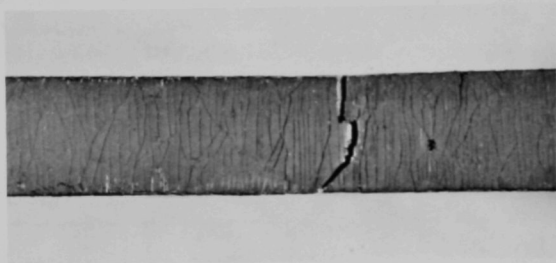
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Figure 5

Separation of Nickel
Plating from Base Alloy
on Extruded Cast Ten-
sile Specimen

Figure 6

Cracks in Nickel Plating
on Extruded Powder Ten-
sile Specimen



EI-797

3X

Grain Size and Hardness Measurements

Grain-size determinations and hardness measurements were made on both unirradiated and irradiated specimens. The results are tabulated in Table VI. Recrystallization and grain growth occurred on the extruded

Table VI

DPH HARDNESS AND GRAIN SIZE OF Ag-15w/oIn-5w/oCd ALLOY SPECIMENS

Designation	Irradiation Temp, °C	DPH Values(a)		ASTM Grain Size(d)
		Range	Average Value	
Extruded cast alloys CR-6A, B, C, & D	Unirradiated	55 to 78	$66 \pm 8^{(b)}$	7
Extruded cast alloys CR-6A, B, C, & D	300	36 to 55	$46 \pm 8^{(c)}$	4 to 5.5
Extruded powder alloys PCR-4-E & H	Unirradiated	87 to 95	$90 \pm 2^{(e)}$	
Extruded powder alloys PCR-4-E & H	300	81 to 100	$92 \pm 6^{(f)}$	

(a) A 1-kg load was applied.

(b) A total of 30 measurements were made.

(c) A total of 9 measurements were made.

(d) As determined by ASTM designation E112.63, 1963 Revision.

(e) A total of 18 measurements were made.

(f) A total of 12 measurements were made.

cast specimens during irradiation. The ASTM grain size of the unirradiated, extruded cast specimens was 7.0. Corresponding postirradiation values ranged from 4 to 5.5.

The DPH values on the unirradiated, extruded cast alloys ranged from 55 to 78, with an average value of 66 and a standard deviation of ± 8 DPH units. After irradiation, the values ranged from 36 to 55, with an average value of 46 and a standard deviation of ± 8 DPH units.

Table VII lists the results of a Knoop hardness survey conducted on specimens of the extruded cast alloy. In general, the measurements indicate that areas near the specimen surface were slightly harder than interior areas. One specimen exhibited an area, 0.1 cm in from the surface of the alloy, that was significantly harder than the surrounding areas. The reason for this localized area of increased hardness is not known.

Table VII
KNOOP HARDNESS SURVEY OF
IRRADIATED Ag-15w/oIn-5w/oCd ALLOYS

Specimen Designation	Irradiation Temp, °C	Knoop Hardness ^(a)	
		Location, cm ^(b)	Values
Extruded cast alloy CR-6A	300	Surface	13.1
		0.025	11.2
		0.050	10.5
		0.075	10.6
		0.100	10.5
Extruded cast alloy CR-6B	300	Surface	11.4
		0.025	10.2
		0.050	8.8
		0.075	8.9
		0.100	10.5, 11.2, 13.1
		0.125	8.7

(a) A 200-g load was applied.

(b) Measurements were made at the surface of each core alloy, and then at 0.025-cm intervals towards the specimen center.

For the extruded powder alloys, the hardness values did not change appreciably with irradiation. The DPH values on the unirradiated, extruded powder alloys ranged from 87 to 95, with an average value of 90 and a standard deviation of ± 2 DPH units. After irradiation, the values ranged from 81 to 100, with an average value of 92 and a standard deviation of ± 6 DPH units.

The irradiated microstructures of the extruded powder alloys indicated recrystallization had occurred during irradiation, resulting in at least a partial loss of the oriented as-fabricated structure.

Metallographic Examination

The absorption of neutrons by the isotopes of the Ag-In-Cd alloy leads to nuclear transformations. The majority of the transformed silver goes to cadmium, the indium to tin, and the cadmium to another isotope of cadmium. There is a limit to the amount of tin that can be dissolved in the alloy. It is anticipated that after extended operation at high power levels, the limit of solubility of tin will be reached and any further production of tin will result in its rejection from solid solution and the formation of a second-phase precipitate. This precipitate may be especially prevalent in the more highly burned-out surface layers of the specimens. The pre- and postirradiation microstructures are compared in Figure 7. The microstructures indicated that the alloys had remained single-phase during irradiation. There was no second phase present, even in the specimen that analyzed 1.5% tin after irradiation.

The metallographic specimens were mounted in Bakelite and ground to a 600 grit finish using silicon carbide papers. The specimens were prepared by alternately polishing with Linde A compound on microcloth and swab-etching with a solution composed of one part stock solution and nine parts water. The stock solution contained $K_2Cr_2O_7$, NaCl, and H_2SO_4 in the ratio of 50/1/5, respectively. The specimens were swab-etched just before examination.

Burnup Analysis

One specimen from each capsule was chemically analyzed for the quantity of silver, indium, and cadmium present. The amount of tin was obtained by difference, noting that the atom fraction of indium before irradiation is equal to the sum of the atom fractions of indium and tin after irradiation. The results of these analyses, when compared with the unirradiated compositions, indicate the elemental burnout during irradiation. Table II lists the pre- and postirradiation compositions.

The specimens were prepared for analysis by dissolving them in nitric acid, and by diluting and cooling the solution. The solution was titrated with standard ammonium thiocyanate solution to determine the amount of silver present. The silver thiocyanate precipitate was washed and filtered, and the filtrate was analyzed for indium and cadmium. The cadmium was analyzed colorimetrically, and the indium by spectrochemical methods.

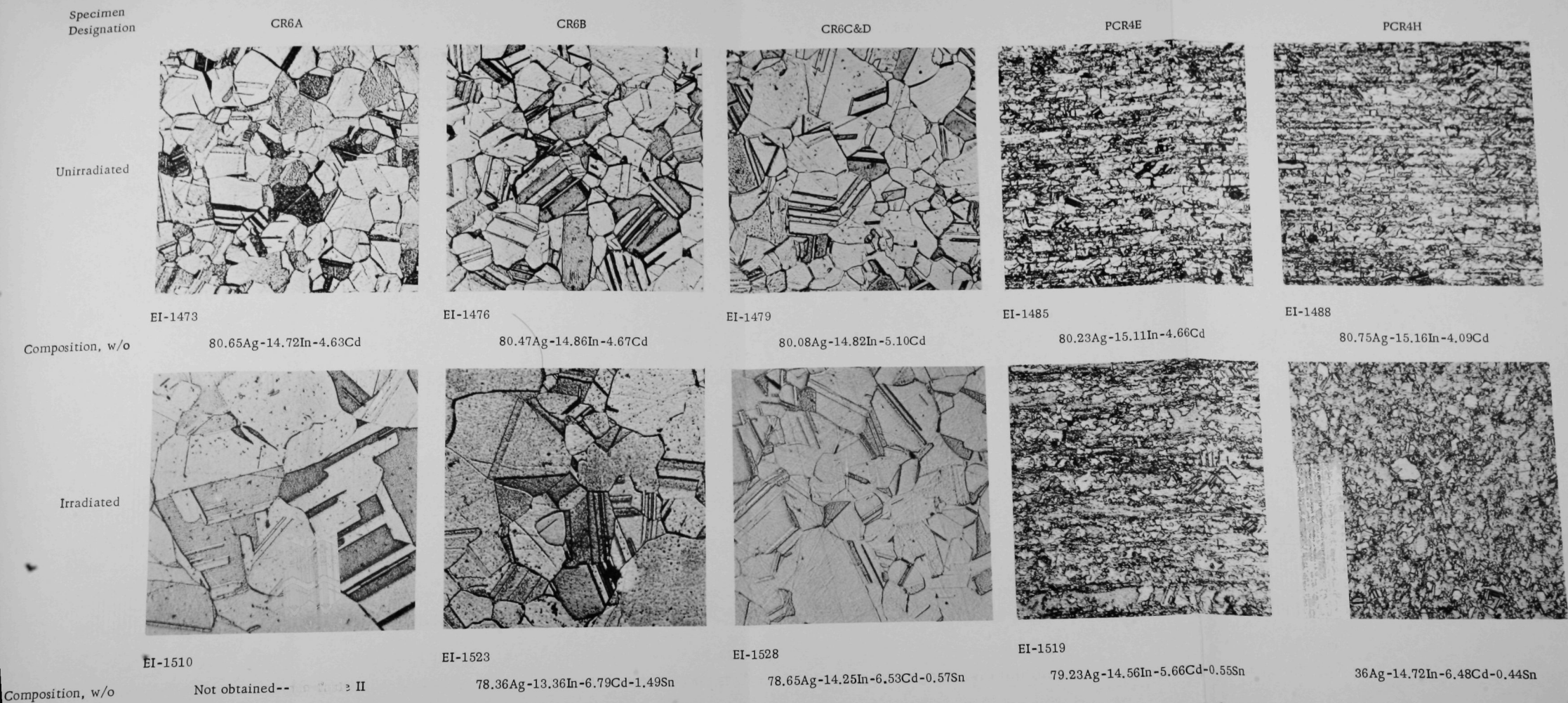


Figure 7. Comparison of Pre- and Postirradiation Microstructures on Ag-15w/oIn-5w/oCd Alloys. All photos are bright field at 250X.

CONCLUSIONS

1. There was very little change in the 0.2% yield and ultimate tensile strength of extruded cast and extruded powder Ag-15w/oIn-5w/oCd alloy specimens irradiated to integrated, unperturbed, thermal neutron fluxes of between 1.0 and 1.7×10^{21} nvt at 300°C .
2. The extruded cast alloys exhibited 0.2% yield strengths ranging from 114 to 140 kg/cm² in the unirradiated condition, and from 96 to 150 kg/cm² after irradiation. The percent elongation and reduction in area in both cases normally ranged from 50 to 60%.
3. The extruded powder alloys exhibited 0.2% yield strengths ranging from 190 to 228 kg/cm² in the unirradiated condition, and from 214 to 242 kg/cm² after irradiation. There was a noticeable improvement in both the percent elongation and reduction in area with irradiation, the postirradiation values being approximately twice the unirradiated values of 10%.
4. Recrystallization and grain growth occurred in all specimens during irradiation.
5. The ASTM grain size on the extruded cast specimens changed from 7 to 4-5.5.
6. The extruded cast specimens softened during irradiation. Unirradiated material had DPH values ranging from 55 to 78, with corresponding values after irradiation of 36 to 55.
7. There was little overall change in the hardness of the extruded powder alloys. Unirradiated material had DPH values ranging from 87 to 95, with corresponding values after irradiation of 81 to 100.
8. The metallographic examinations indicated that the microstructures had remained single-phase during irradiation.

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-- Specimen fabrication, capsule irradiations, preirradiation data

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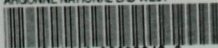
-- Postirradiation data collection

- | | |
|---|--------------------|
| R. W. Bane and J. P. Faris | -- Burnup analyses |
| R. Carlander, W. A. Ahrens, and W. C. Kettman | -- Metallography |

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